

NASA Technical Paper 1156



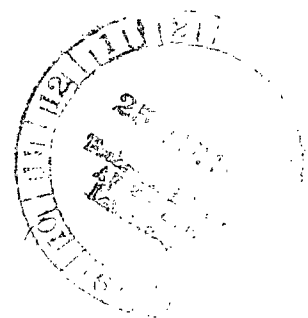
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Friction and Wear of Radiofrequency-Sputtered Borides, Silicides, and Carbides

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FRICION AND WEAR OF RADIOFREQUENCY-SPUTTERED BORIDES, SILICIDES, AND CARBIDES

by William A. Brainard and Donald R. Wheeler

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SUMMARY

The friction and wear properties of several hard, refractory compounds in coating form were examined. The compounds were titanium boride (TiB_2), molybdenum boride (Mo_2B_5), titanium silicide (TiSi_2), molybdenum silicide (MoSi_2), titanium carbide (TiC), molybdenum carbide (Mo_2C), and boron carbide (B_4C). The coatings were applied by radiofrequency (rf) sputtering to AISI type 440C bearing steel substrates. Testing was done with a pin-on-disk wear apparatus at loads from 0.1 to 5.0 newtons at sliding speeds of 25 centimeters per second.

The carbides in general, and titanium carbide in particular, provided the best friction and wear protection. At loads up to 1.0 newton, titanium boride coatings exhibited low wear and friction. Molybdenum boride coatings failed due to spalling at loads in excess of 0.1 newton. Titanium silicide coatings were poor even at the lightest loads (0.1 N). Although tenaciously adherent, molybdenum silicide coatings yielded high friction and high rider wear, with metallic transfer.

Preoxidizing the 440C steel surface prior to coating improved coating adherence so that the film could be tested to higher loads before it spalled. This improvement is related to the better bonding of the refractory compound to the iron oxide surface that was formed by preoxidizing the 440C steel.

INTRODUCTION

Hard facings are commonly used to improve wear resistance. Hardened surfaces are routinely prepared by conventional techniques such as carbonizing or nitriding. Hard coatings are applied by electroplating, welding, or plasma spraying (ref. 1). These methods of application are quite suitable for some substrates and coating materials, but not for all. Limitations imposed by the high temperatures required for

furnace or spraying methods preclude their use on some components or alloy systems. Oxidation of the coating material and residual mechanical stresses induced in components by the thermal gradient are other considerations. Many of these limitations are avoided when coatings are applied by radiofrequency (rf) sputtering (ref. 2). Sputtering has been found useful for the deposition of the hard, refractory compounds that are of current interest as wear-resistant coatings (ref. 3).

The refractory metal borides, silicides, and carbides are of interest because of their extreme hardness (up to 5000 kg/mm^2) and their potential for use at high temperatures. Previous work on refractory compounds suggested their potential usefulness as antiwear coatings when applied by rf sputtering (ref. 3). However, data obtained by X-ray photoelectron spectroscopy (refs. 4 and 5) showed that wide variations in coating composition were obtained, depending on the deposition condition, and that care must be taken with regard to each specific compound in order to obtain maximum coating adherence and composition properties.

This investigation was conducted to determine the friction and wear characteristics of sputtered refractory borides, silicides, and carbides. These compounds had been deposited under conditions determined from previous work (refs. 4 and 5) in order to produce coatings close to target composition with a minimum of oxide impurities and with improved adherence to the steel substrate. The compounds investigated were titanium silicide (TiSi_2), molybdenum silicide (MoSi_2), titanium boride (TiB_2), molybdenum boride (Mo_2B_5), titanium carbide (TiC), molybdenum carbide (Mo_2C), and boron carbide (B_4C). The friction and wear properties of these films as coatings on type 440C bearing steel were determined by using a pin-on-disk friction and wear device.

APPARATUS AND PROCEDURE

Radiofrequency Sputtering

The sputtering of the materials used in this investigation was done in a commercial radiofrequency diode apparatus operating at 13.56 megahertz. The apparatus is shown schematically in figure 1. The material to be sputter deposited is in the form of a hot-pressed, disk-shaped compact, 15.2 centimeters in diameter, that was commercially purchased. The compact, or target as it is called when mounted, is cemented with a silver conductive epoxy onto a copper backing plate (0.60 cm thick). The copper backing plate is mounted onto a water-cooled rf electrode also 15.2 centimeters in diameter. The specimen to be coated is placed 2.5 centimeters directly below the target on an electrically insulated block. The insulated block sets on the grounded substrate table. (The target and the grounded table constitute the rf diode.) An additional voltage from 0 to -1500 volts dc may be applied directly to the specimen

either for specimen cleaning by direct-current sputter etching or for biasing the specimen during film deposition and growth.

The entire substrate table can be rotated so that the disk specimen may be moved out from under the target. The target can thus be cleaned without contaminating the specimen. If desired, the disk could be cleaned by sputter etching during this time.

The entire diode system is contained in a glass bell jar, 45 centimeters in diameter. The system is mechanically forepumped and oil diffusion pumped through a liquid-nitrogen-cooled baffle. During deposition, high-purity argon (99.9995 percent) was bled continuously into the system through a leak valve. A dynamic pressure balance between the pumping system and the argon leak of approximately 2.7 N/m^2 ($20 \text{ } \mu\text{m}$) was maintained in the bell jar.

Before deposition, the target was cleaned by rf sputtering until little pressure rise due to outgassing occurred. Then the selected bias voltage was set, the specimen was rotated back under the target, and deposition was begun. The power density was constant for all coatings at 1.64 W/cm^2 . Higher power levels increased contamination by heating of the target (ref. 3). Deposition times were from 20 to 60 minutes, depending on the material, which produced coatings from 200 to 300 nanometers thick for all materials, as determined by surface profilometer measurements of a step. All substrates were biased during coating deposition. The bias voltages were -300 volts for the carbides and borides and -100 volts for the silicides. The use of bias voltages was based on an earlier work (ref. 5).

Friction and Wear Testing

The rf-sputtered films were tested on a pin-on-disk apparatus. The pin-on-disk configuration is often used for solid film lubrication testing. The apparatus shown in figure 2 consists of a flat 6.4-centimeter-diameter disk that is mounted on the end of a rotating shaft. The disk specimen is a type 440C bearing steel disk that has been rf sputter coated with one of the test materials. Loaded against the surface of the disk is a 0.476-centimeter-radius pin of 304 stainless steel. The pin is mounted in a holder on the end of, and perpendicular to, a gimbal-supported arm. The pin is loaded against the surface of the disk by weights hung on the arm halfway between the pin and the gimbal. Normal loads from 0.1 to 50 newtons (10 to 500 grams force) were used. The end of the arm opposite the pin holder is attached to a strain-gage bridge, which measures the frictional force. A new pin specimen was used for each test.

The entire apparatus is enclosed in a clear plastic box. A constant flow of dry nitrogen ($<20\text{-ppm H}_2\text{O}$) is maintained in the box prior to and during friction tests in order to minimize environmental effects.

The experiments reported herein were each conducted for 30 minutes at a constant speed of 25 centimeters per second. After the friction testing, the disks were examined by optical microscopy and surface profilometry to determine the extent, if any, of film wear or spalling from the 440C substrate. Film spalling was evident on the surface profile tracing by the appearance of sharp, vertical-sided, flat-bottomed cross sections, which are characteristic of a brittle film spall.

The pin-on-disk configuration provides extremely high contact stresses in contrast to the conforming type of contact. Because of the uncertainties with regard to modulus values for coated substrates, no attempts to calculate Hertzian stresses were made for the coated sample. However, the Hertzian stress for the uncoated disk contacted by the 304 stainless steel pin at 0.5-newton load was 24.65 kg/mm^2 . Any coating that performs well under such extremes would probably perform as well or better under less severe conditions. Thus, in this respect, the pin-on-disk test can be regarded as an upper limit screening test for these coatings.

Specimen Preparation

The 440C bearing-steel disk and 304 stainless-steel pin specimens were all mechanically polished in the same manner before they were coated. The pins and disks were abraded on silicon carbide paper down to 600 grit. Then they were polished with 3-micrometer diamond paste and lapped with 1-micrometer alumina. After lapping, the disks were rinsed with alcohol and dried and, where required, were inserted into a muffle furnace for deliberate oxidizing. Previous work (ref. 5) had shown that coating adherence was significantly improved if the 440C surface was oxidized prior to coating. The disks were heated in air for 20 hours at 340°C and then allowed to slowly cool. The oxide formed at this temperature and for this time may not be the optimum for improving adherence; however, these conditions provided a reasonable specimen preparation time and a basis by which the coatings could be compared. A detailed study of disk oxidation conditions is probably required individually for each of the coating materials; however, such a study was beyond the scope of this work.

Refractory Compounds

The properties of the refractory compounds examined in this investigation are summarized in table I. When a spread in literature values was found, all values are reported rather than an average of the data.

RESULTS

Borides

Figure 3 is a summary of the friction performance of sputtered boride coatings. The open symbols denote data taken when the disk specimen was not preoxidized but had been subjected to sputter etch cleaning (~ 1200 V for 15 min) prior to coating.

All boride coatings were deposited with a bias voltage (~ 300 V) that was selected to provide the optimum in stoichiometry and the minimum in impurities based on the work reported in reference 4. A reference line on the figure indicates the approximate value for a 304 steel rider sliding on uncoated 440C. The level is extrapolated from friction tests on uncoated samples at loads of 0.5 to 2.0 newtons. When film failure occurs (i.e., the film spalls and separates from the substrate), the friction should tend toward the values associated with uncoated samples.

For Mo_2B_5 coatings, tests with both oxidized and nonoxidized (sputter etched) samples at 0.1-newton load showed low friction (average friction coefficient $\bar{\mu} < 0.2$). However, both coatings failed at a higher load of 0.25 newton. Oxidizing the 440C substrate apparently provided little improvement in adherence of this particular boride.

In contrast, the friction properties of TiB_2 coatings were significantly improved by oxidizing the disk because of greater coating adherence. TiB_2 coatings showed a slight decrease in friction with load to 0.5 newton. At 1.0 newton the coating started to spall, with friction approaching that of the uncoated 440C.

Silicides

Data for the friction behavior of the TiSi_2 and MoSi_2 coatings are presented in figure 4. For both materials, even at the lightest load, the friction value was that of an uncoated sample because of the rapid and total failure of film by spalling. Film failure was verified by subsequent surface profile examination of the wear track, which showed a sharp rectangular-shaped wear track in cross section. Oxidizing the 440C substrate produced no improvement in adherence for TiSi_2 but considerably improved the adherence of MoSi_2 . MoSi_2 films on oxidized 440C were so tenaciously bonded that metallic transfer from the 304 steel rider was observed on the coating wear track after testing (fig. 5). The MoSi_2 coating exhibited higher friction than either of the boride coatings at 0.1 newton, but this higher friction is associated with the intrinsic frictional properties of the coating rather than with film spalling and subsequent metal-to-metal contact. This was the only coating material that exhibited adhesive transfer of the metallic rider material.

Carbides

Friction data for the three carbide sputter coatings are presented in figure 6. The carbides, as a class, were the most adherent to 440C when oxidized. As with the borides and silicides, all the carbide coatings were sputtered with an imposed bias voltage in order to optimize bulk film quality. Note that the load scale for the carbide extends to five times the loads in figures 3 and 4. Boron carbide coatings, although adhering well up to 2.0 newtons, exhibited very high friction, $0.8 < \bar{\mu} < 0.9$.

Surface profile traces at a vertical magnification of 20 000 showed, however, minimal coating wear up to 1.0-newton load. In an application where high friction is either desirable or not objectionable, B_4C might be of use. Mo_2C coatings exhibited low friction at the lightest loads, but the friction increased as the load increased to 2.0 newtons, the maximum tested, because segments of the wear track experienced some spalling. Even at 2.0 newtons, the film was still partially intact. Titanium carbide was clearly superior to the other carbides in friction and adherence even at loads of 5.0 newtons. After testing at 5.0 newtons, surface profile examination showed the coating still intact, with no spalling along the entire wear track.

DISCUSSION

For comparison of all seven materials, the rider wear volumes and friction coefficients are summarized in figure 7. All data are for the same load of 0.25 newton.

It is, of course, recognized that friction and wear are separate measurements; one does not necessarily reflect the other. High wear does not mean high friction, nor does low friction necessarily correlate with low wear. The application of these coatings will generally be for wear or abrasion resistance. Friction property may be of little consequence in many applications but an important consideration in others.

A coating of almost any of the refractory silicides, borides, or carbides should improve the wear resistance of a metallic part. From table I, it can be seen that, with the possible exception of $TiSi_2$, all the compounds are significantly harder than 440C ($\sim 700 \text{ kg/mm}^2$). Since the wear rate is inversely proportional to hardness, both in abrasive wear and in adhesive wear (ref. 6), any of these compounds can be expected to improve wear resistance if they remain bonded to the surface. On the other hand, should the film fail to adhere, the film debris would be abrasive and would cause severe wear.

Adherence, generally, is improved by the presence of an oxidized layer on the surface of the 440C prior to coating. Since 440C is an alloy composed primarily of iron (80.1 wt %) and chromium (17.3 wt %), it is of interest to know which alloy constituent is most involved in the improved adherence after oxidation. To determine this,

samples of pure iron and pure chromium were subjected to the same furnace treatment as the 440C disks. After the furnace treatment, these samples, along with iron and chromium samples that had not been furnace treated and that were cleaned by sputter etching, were sputter coated with molybdenum carbide. Molybdenum carbide was used because it adhered significantly better to oxidized 440C than any of the other coatings.

Figure 8 shows micrographs of the Mo_2C coatings on the sputter-etched iron and chromium samples. The film is intact on the iron surface but peels off spontaneously on the chromium. The chromium samples that had been furnace treated had a similar appearance: the Mo_2C film pulled off because of the intrinsic film stresses introduced into the film during deposition. The preoxidized iron sample, on the other hand, showed a significant change in appearance (fig. 9). The Mo_2C coating on the preoxidized iron sample was darker and very textured in appearance, suggesting a reaction between the film and the substrate. The preoxidized surface did not have this appearance prior to coating. This reaction undoubtedly is responsible for the improvement in film adherence on oxidized 440C.

An analysis of an oxidized 440C disk by Auger emission spectroscopy, shown in figure 10, verifies that the surface is primarily iron oxide. Very little chromium is observed. The importance of oxidized layers on metal surfaces in bonding is not new. Research on cermets (ref. 11) as early as 1949 recognized that bonding was promoted by mixing of oxides. Further, the electronics industry recognized the improvements to be gained in ceramic-metal bonding by oxide mixing and/or spinel formation (refs. 12 and 13).

It, of course, must be emphasized that the results reported for these materials are unique to the material combinations used for the disk and rider. The fact that bonding is promoted by the formation of iron oxide suggests that these results would generalize to the coating of most steel surfaces, where iron oxides would be expected to form. For nickel-base alloys or austenitic stainless steels, different coating-substrate interactions must be considered.

SUMMARY OF RESULTS

Friction and wear tests on radiofrequency-sputtered coatings of refractory borides, silicides, and carbides yielded the following results:

1. The carbides in general, and titanium carbide in particular, provided the best wear performance. Titanium carbide coatings also exhibited low friction.

2. At lighter loads (<1.0 N), titanium boride coatings showed good wear properties and very low friction, but the molybdenum boride coatings failed by spalling at loads beyond 0.1 newton.

3. Titanium silicide coatings were poor at even the lightest load (0.1 N). Although it remained adherent, the molybdenum silicide coating yielded high friction and high rider wear with metallic transfer.

4. Preoxidizing the 440C substrate, prior to coating, improved coating adherence, prevented spalling, and thus extended the useful load range for all compounds except titanium silicide and molybdenum boride.

5. The improvement in adherence on preoxidized surfaces is related to better bonding between the coating and the iron oxide film formed on the 440C.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, January 12, 1978,
506-16.

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TABLE I. - PROPERTIES OF REFRACTORY COMPOUNDS

Compound	Melting point, °C	Density g/cm ³	Crystal structure	Free energy of formation, ΔH_f^0	Microhardness, kg/mm ² (at load, g)	Elastic modulus, kg/mm ²	Shear strength, kg/mm ²	Porosity, percent	Coefficient of expansion, per °C
TiSi ₂	^a 1460-1540	^a 4.39 ^d 4.15	Orthorhombic ^b	^b 133 ^a 179	^b 618 (100) ^a 892 (50)	^a 26 400	^a 21.0	(c)	^a 10.5×10 ⁶ ^d 10.4
MoSi ₂	^a 2030	^a 5.9-6.3	Tetragonal ^b	^b 108	^b 1290 (100) ^a 1200 (50) ^e 1259 (100)	^b 38 400 ^a 43 000 ^d 35 150	^a 35.1	(c)	^b 8.4×10 ⁶ ^a 8.25 ^d 8.1
TiB ₂	^a 2980 ^e 2600	^a 4.5	Hexagonal ^a	^b 150 ^a 183	^b 3480 (50) ^a 3370 (30)	^b 37 400 ^a 54 000 ^d 43 600	^a 24.5	1	^b 6.4×10 ⁶ ^a 5.5 ^d 8.6
Mo ₂ B ₅	^b 2300 ^a 2100	^a 7.0	Rhombohedral Hexagonal ^a	^b 131	^b 3220 (50) ^d 3000 (100)	-----	^a 17.5-35.1	10-35	^d 5.0×10 ⁶
TiC	^a 3147 ^e 3250	^a 4.9	Cubic ^b	^b 115	^b 3200 (50) ^a 3000 (100) ^d 3200 (100)	^b 32 000 ^a 46 000 ^e 42 200 ^d 37 300	^a 87	0	^b 7.42×10 ⁶ ^a 7.74 ^e 5.5
MoC ₂	^a 2410 ^c 2690	^b 9.2 ^a 8.9	Hexagonal ^b	^b 11	^b 1950 (50) ^a 1499 (30) ^e 1460-1800 (c)	^b 22 100 ^a 54 400 ^e 22 076	^a 5.0	27	^b 4.4×10 ⁶ ^a 7.8 ^d 6.7
B ₄ C	^e 2450	^e 2.51	Rhombohedral ^a Hexagonal ^e	----	^a 4950 (30)	^b 44 800 ^e 45 700	^a 31	(c)	^e 4.5×10 ⁶

^aRef. 7.^bRef. 8.^cValue uncertain.^dRef. 9.^eRef. 10.

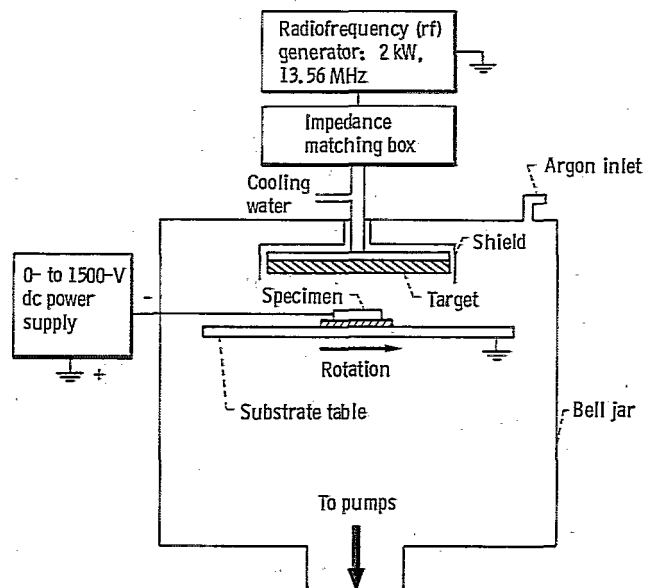


Figure 1. - Schematic of radiofrequency sputtering apparatus.

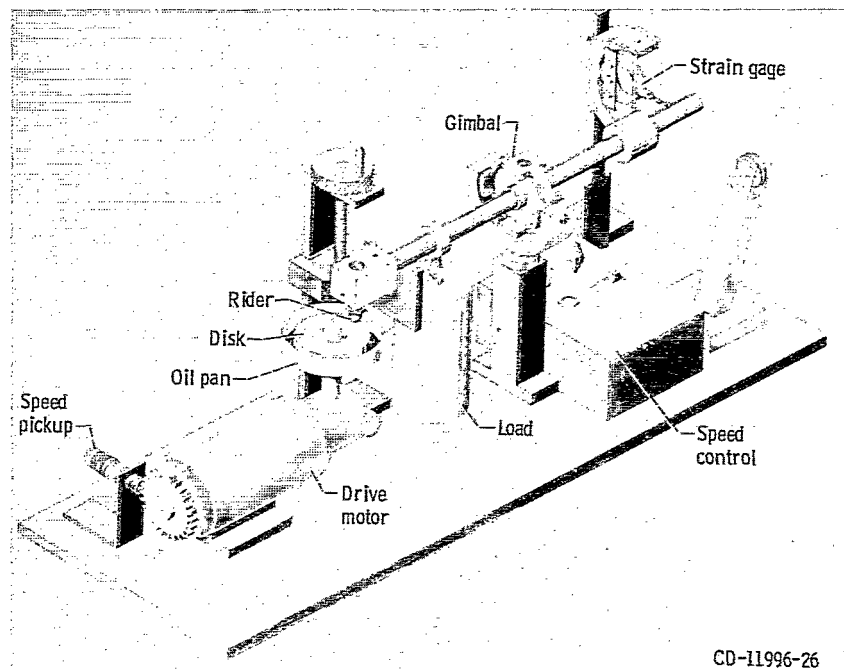


Figure 2. - Pin-on-disk friction and wear apparatus.

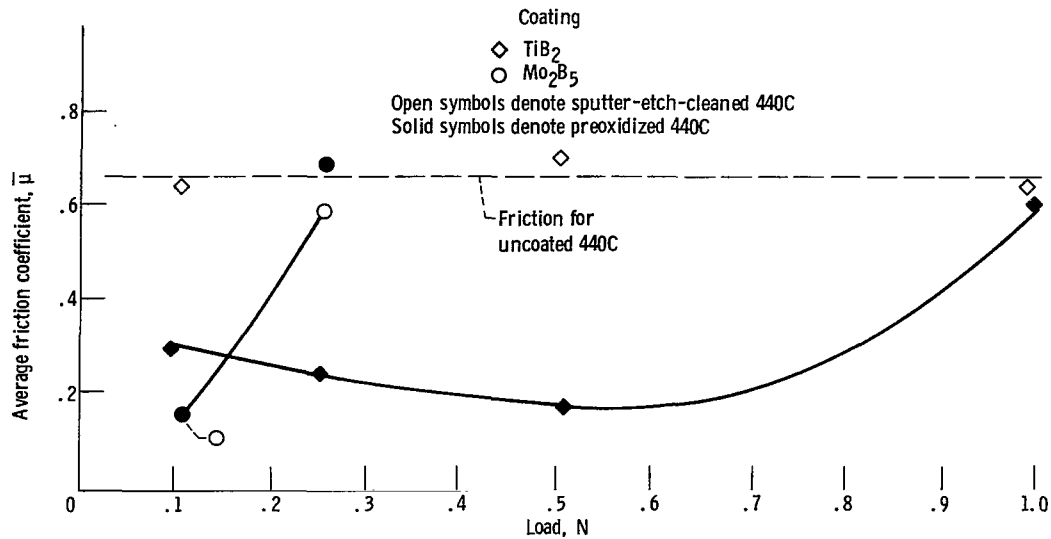


Figure 3. - Friction coefficient as a function of load for radiofrequency sputter-deposited boride coatings on 440C steel disks. Bias voltage, -300 V; rider, AISI 304 stainless steel; atmosphere, gaseous nitrogen; sliding speed, 25 cm/sec.

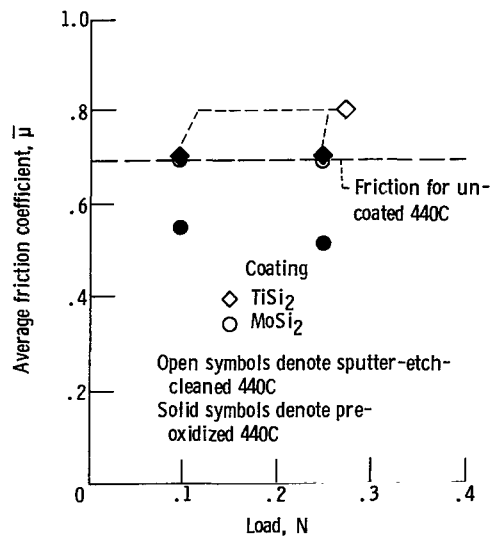


Figure 4. - Friction coefficient as a function of load for radiofrequency sputter-deposited silicide coatings on 440C steel disks. Bias voltage, -100 V; rider, AISI 304 stainless steel; atmosphere, gaseous nitrogen; sliding speed, 25 cm/sec.

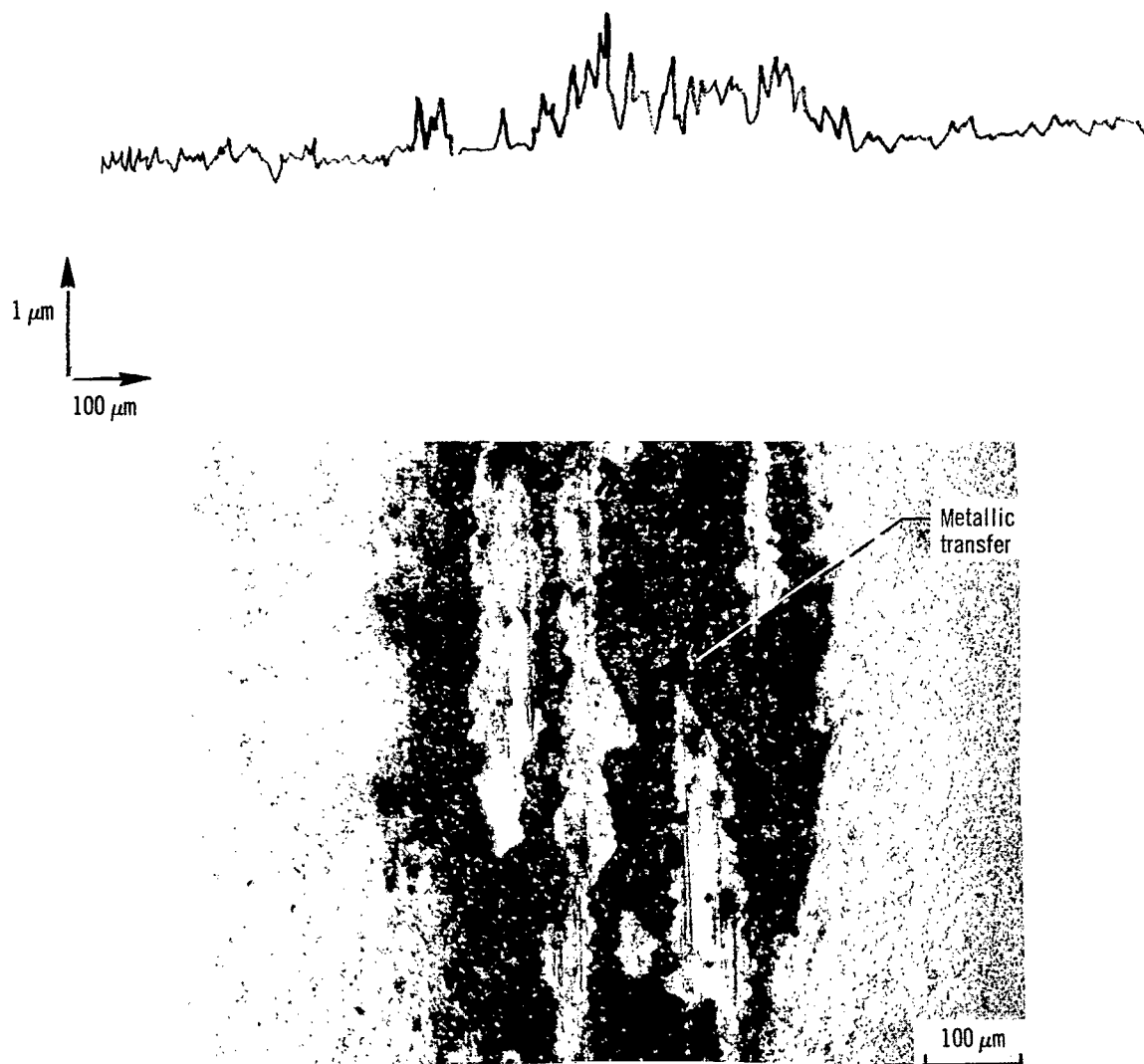


Figure 5. - Surface profile trace and photomicrograph of wear track on oxidized 440C steel disk radiofrequency sputter coated with molybdenum silicide. Bias voltage, -100 V ; load, 0.1 N ; sliding speed, 25 cm/sec .

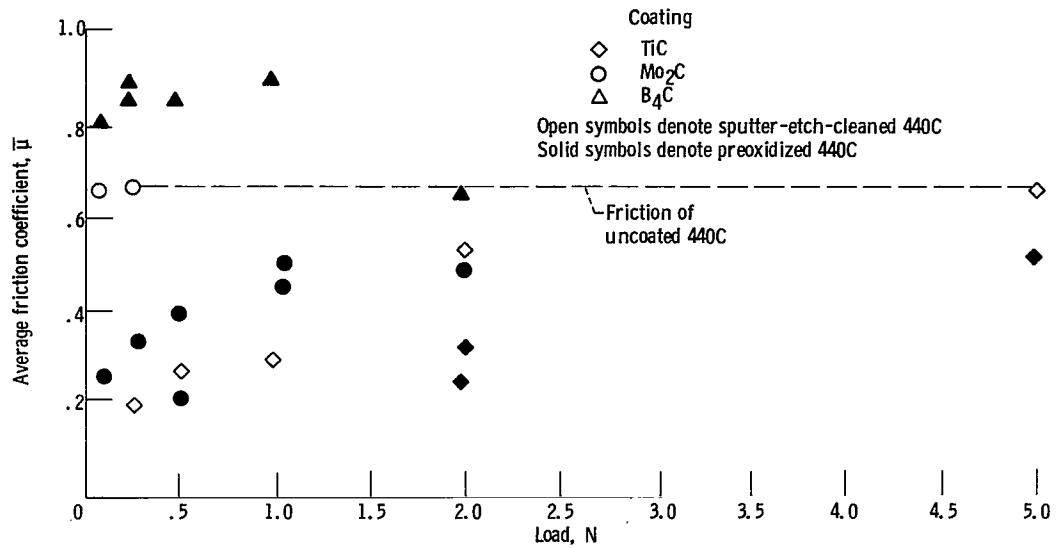


Figure 6. - Friction coefficient as a function of load for radiofrequency sputter-deposited carbide coatings on 440C steel disks. Bias voltage, -300 V; rider, AISI 304 stainless steel; atmosphere, gaseous nitrogen; sliding speed, 25 cm/sec.

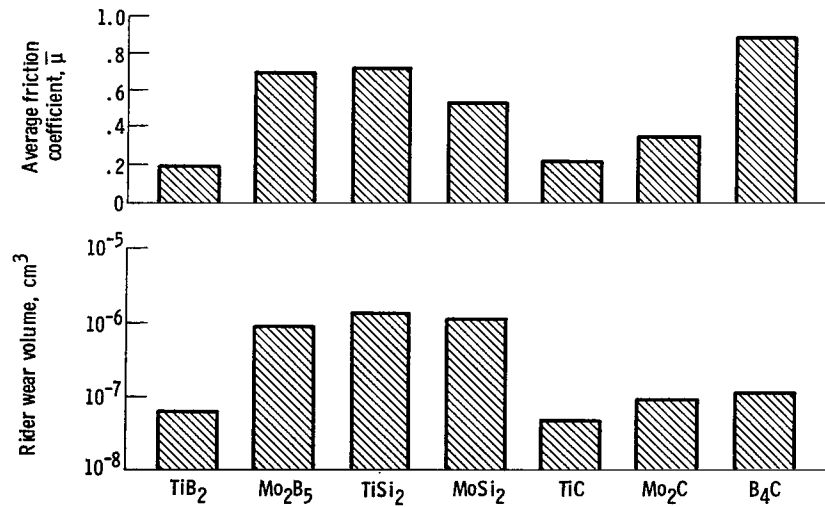
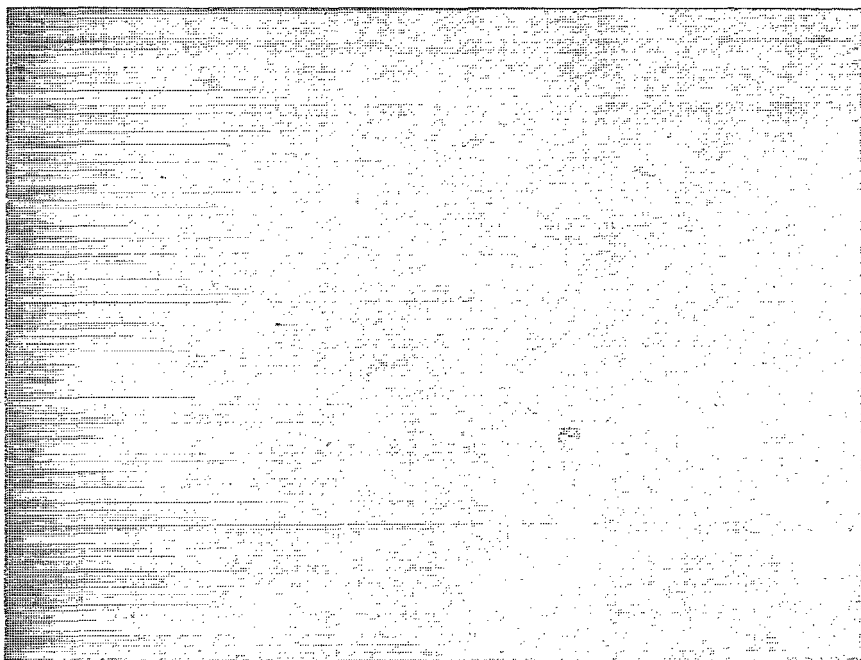


Figure 7. - Friction coefficient and rider wear caused by AISI 304 stainless-steel rider sliding on various refractory metal compounds radiofrequency sputter coated on oxidized 440C steel disks. Load, 0.25 N; atmosphere, gaseous nitrogen; sliding speed, 25 cm/sec; duration, 30 min.



(a) Iron.



(b) Chromium.

Figure 8. - Photomicrographs of sputter-etch-cleaned iron and chromium radiofrequency sputter coated with molybdenum carbide. Bias voltage, -300 V.

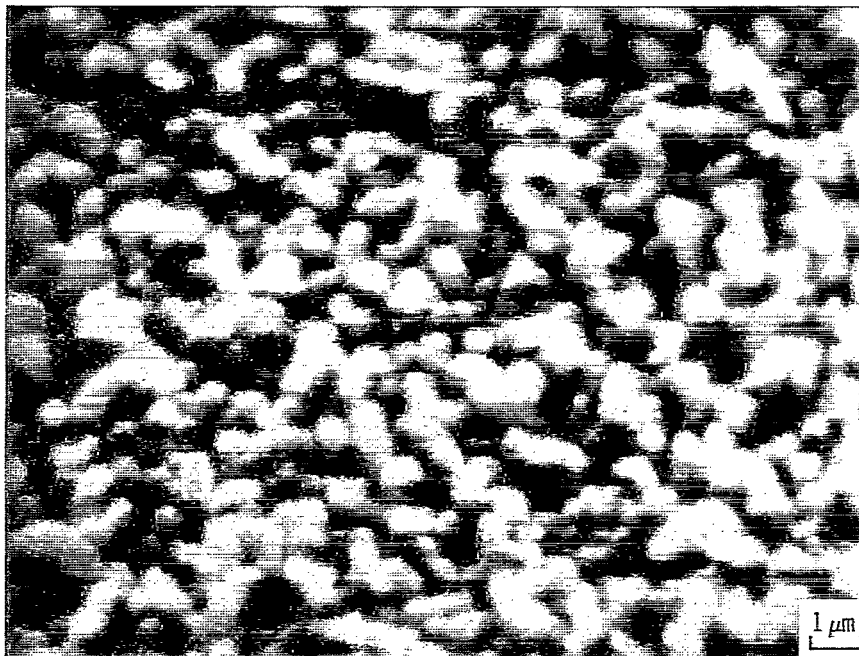


Figure 9. - Photomicrograph of preoxidized iron radiofrequency sputter coated with molybdenum carbide. Bias voltage, -300 V.

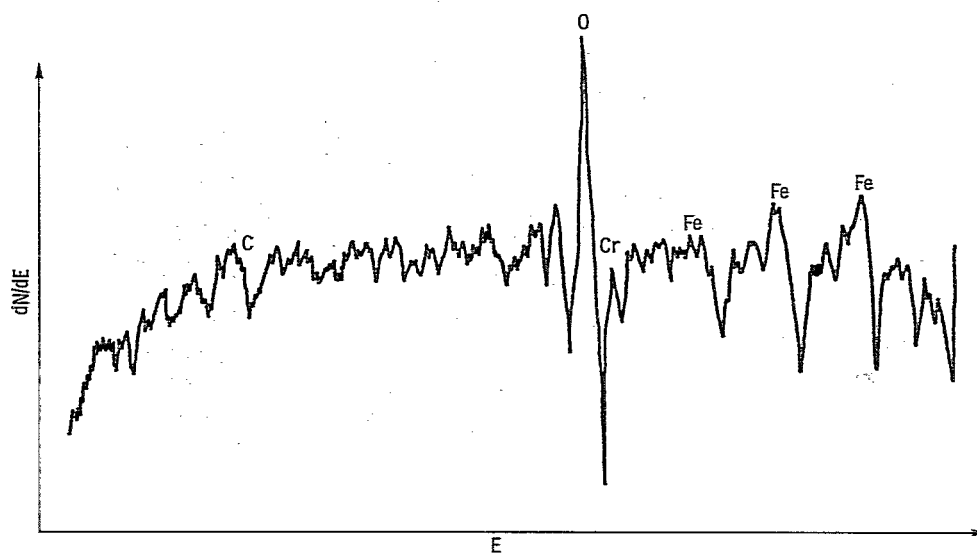


Figure 10. - Auger spectrum of oxidized 440C steel disk. Furnace temperature, 340⁰ C; duration, 20 hr.

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16. Abstract <p>The friction and wear properties of several refractory compound coatings were examined. These compounds were applied to 440C bearing steel surfaces by radiofrequency (rf) sputtering. The refractory compounds were the titanium and molybdenum borides; the titanium and molybdenum silicides; and the titanium, molybdenum, and boron carbides. Friction testing was done with a pin-on-disk wear apparatus at loads from 0.1 to 5.0 newtons. Generally, the best wear properties were obtained when the coatings were bias sputtered onto 440C disks that had been pre-oxidized. Adherence was improved because of the better bonding of the coatings to the iron oxide formed during preoxidation. As a class the carbides provided wear protection to the highest loads. Titanium boride coatings provided low friction and good wear properties to moderate loads.</p>		
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